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CARIBBEAN RESEARCH INSTITUTE

NASA Grant #NGR-52-083-002

to the

College of the Virgin Islands

for

Research in the Optimization of Separator Sub-systems

for GC/MS Life Detection Instrumentation

ANNUAL REPORT

November 15, 1967 - December 15, 1968

Frank B. Gray, Project Director

Caribbean Research Institute Project #29

ANNUAL REPORT

1967 - 1968

NASA Grant #NGR-52-083-002

CRI Project #29

I. Initial Phase Activities. Two major activities were carried out during the initial phases of the above grant.

A. Contacts with NASA Supported Laboratories and with Equipment Suppliers in the GC/MS Field.

Personal contacts were made and maintained with other laboratories carrying on research in the GC/MS field. These included:

Yale University -- Dr. S. R. Lipsky

The subjects discussed were GC equipment, separators and MS equipment. Specifications were provided for the GC columns currently used in his laboratory for the work on Life

Detection problems. Details of construction of the Lipsky separator were provided and experiences of other laboratories in the area with the Finnigan 1015 were given. It was concluded that the use of a quadrapole MS -- like the Finnigan -- would be most advantageous to the project.

At a later date the loan of a 10 MV GC recorder, Lipsky separator components, columns, column materials and standard C_{12}-C_{18} samples was arranged. These are in current use on the project.

University of Houston -- Dr. Jean Oro; William Updegrove

The subjects discussed were MS equipment, separators and special GC columns. It was their opinion that the quadrapole MS offered many advantages such as sensitivity, stability and broad latitude of pressure-flow input conditions over other types. In addition, some

of their new column designs, such as those with etched internal surfaces, could possibly be fed directly into quadrapole MS giving comparable sensitivities to standard columns used with currently available separators.

J. P. L. -- Charles Giffin; Harold Kruger

The subjects discussed were separators and separator test equipment. The need for a well organized program was agreed upon and J. P. L. designs for certain test equipment were offered.

A concurrent survey was made of possible suppliers of GC/MS equipment and components.

B. Design and Procurement of Basic Instrumentation

From past knowledge of the field and from information obtained through the above contacts it was determined that the purchase of a complete system with a built-in separator test

chamber provided much more flexibility and long range economy than the assembly of a group of individual components. A quotation on such a system was obtained from Finnigan Instruments after a further survey of suppliers in this field. It was also determined that this equipment could be provided on a lease/ purchase basis and NASA Property agreed that the initial payment could come from the current year's procurement funds.

In the design worked out with Finnigan before the order was placed, they agreed to supply, at the standard cost of the "GC Extra," the GC (A) and Separator Test (B) compartments completely equipped as detailed on College of the Virgin Islands Dwg. CRI #29-1 and Figure 1-A. This design produces a minimum amount of "peak spreading" since the GC column, separator and MS inlet are grouped together but still have individual temperature

controls.

The instrument was delivered in mid-September and has been operating in a routine manner since that time.

II. The last quarter's efforts have been directed toward a thorough check-out of the instrumentation, setting up calibration standards and the assembly of a Lipsky separator for initial tests.

The performance of the Finnigan instrument has exceeded, in every detail, our expectations. It has extreme stability, reproducibility and sensitivity. It is quite simple to operate and is provided with many fail-safe features to protect the entire system in case of power or cooling water failures.

Figure 2 is a sample record of a gas chromatogram of a C_6-C_{12}-C_{14}-C_{16} mixture (with individual concentrations in the 10 ugm range) and Figures 3 and 4 are mass spectrograms (10-250 mass range)

of the separated components. The GC separation was accomplished with a 6 ft. column, 1/8 inch I.D. containing 3% OV-17 on 80 mesh chromosorb G, employing He as a carrier gas at 30# pressure and utilizing a 10/1 splitter.

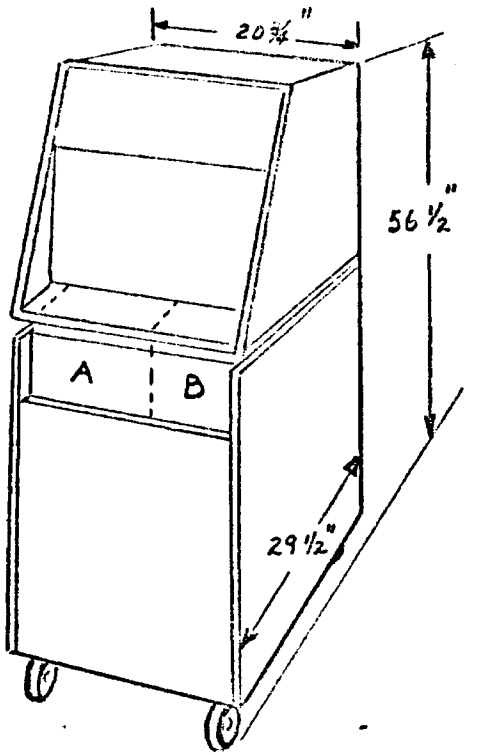
Calibrated samples in the 0.1, 1.0 and 10.0 ugm regions of C_{12} (Ethyl laurate) in C_8 (Xylene) have been made up for the program in separator testing. In addition, pure samples of C_{12}, C_{14} and C_{16} compounds have been ordered for preparation of additional calibration samples. Figure 5 shows two repeat runs of the ability of the instrument to separate the C_8 and C_{12} fractions from a mixture in which the C_{12} concentration was approximately 10 ugm. Figure 6 shows the mass spectrograms taken at the maxima of the GC peaks in Figure 5.

A Lipsky separator employing 140 cm of 0.05 x 0.025 cm teflon tubing has been assembled. Figure 1-A shows the separator with its stainless connecting tubing attached mounted in the separator

test compartment (Det. B - CVI Dwg. #CRI-29). Figure 1-A also shows the physical arrangement of the components in the GC compartment.

Figure 1-B shows the initial assembly step in making up the separator.

Tests have been started on this separator, but have been held up by leaks at the points where the stainless tubing is connected to the teflon tubing. Since the thirteen month period of the initial grant has expired, work on the instrumentation has been halted to prepare this final report. New sealing techniques will be tried on the stainless-teflon joints, in order that the test program may continue when the renewal of the grant is accomplished. Preliminary discussions have been held with J. P. L. (Charles Giffin and Steve Szermay) on making this test program a joint CRI/JPL effort.



Vacuum Console

Dimensions - As above

Modifications -(By Finnigan) Heater - Fan - Heated Syringe Injector Temperature Control

1. Add Compartment A - Install - G.C. Column Splitter Glass Inlet Connection

2. Add Compartment B - Install - Heater - Fan Temperature Control

Electronics Console

Dimensions - As above

Modifications - None

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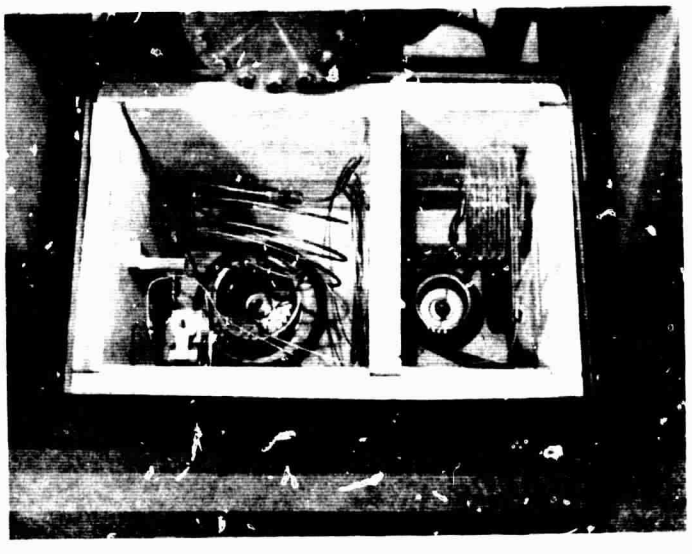
N.C. C.R.I. #29-1 10-2

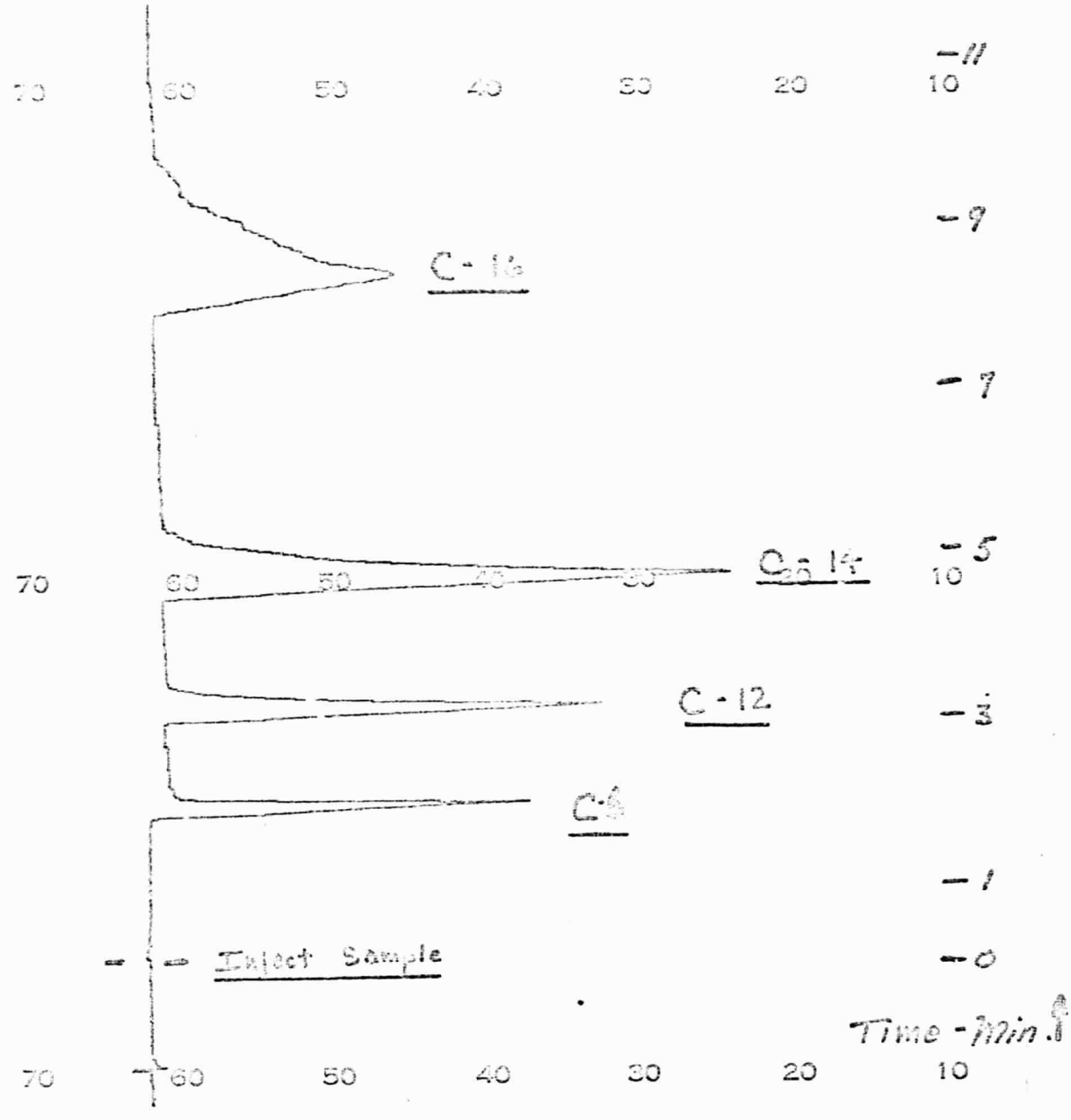
Finnigan #1015 Modifications



A
C G and Separator Test Compartments

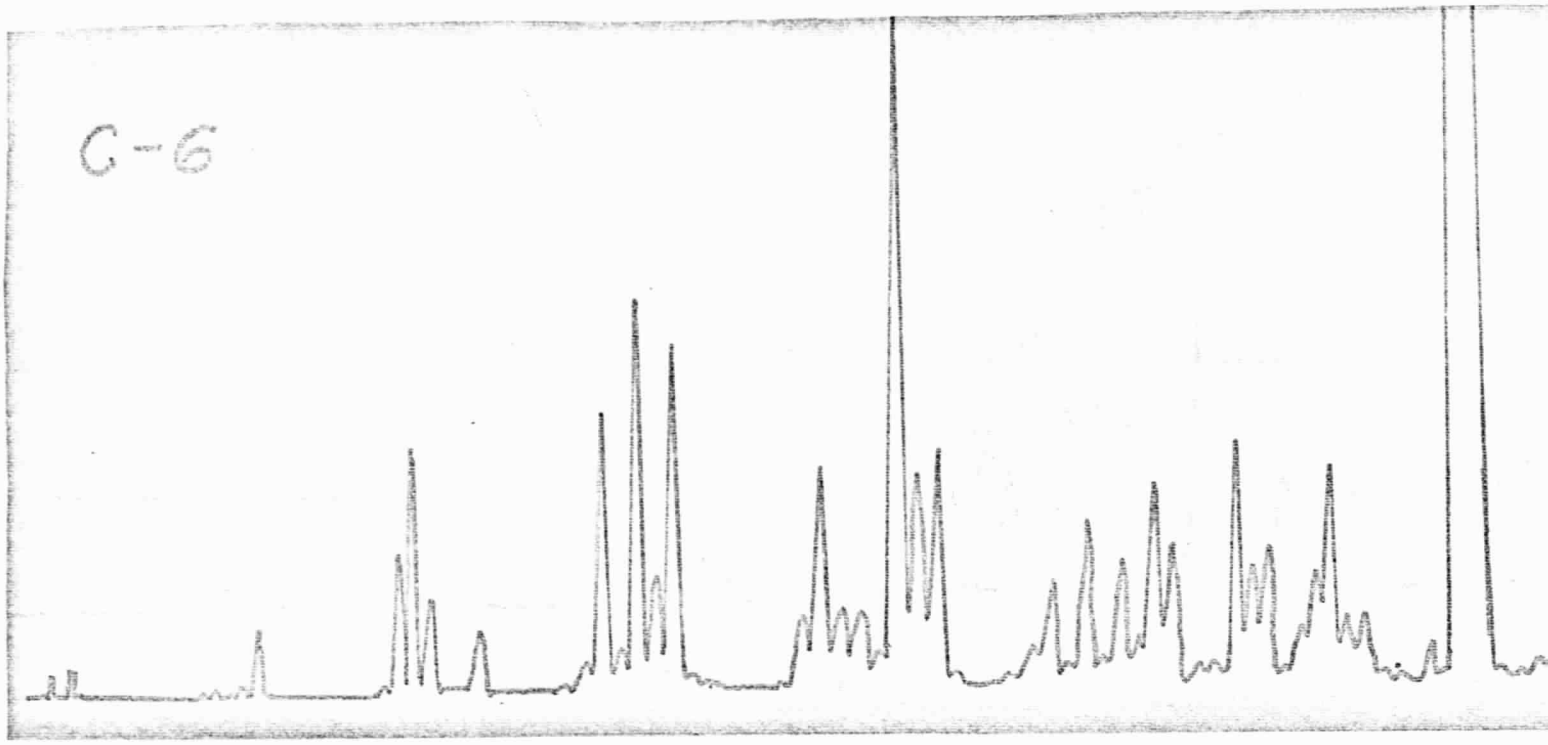
B
Lipsky Separator

Figure 1.



GAS CHROMATOGRAM OF SAMPLE CONTAINING C-6, C-12, C-14, & C-16

Figure 2



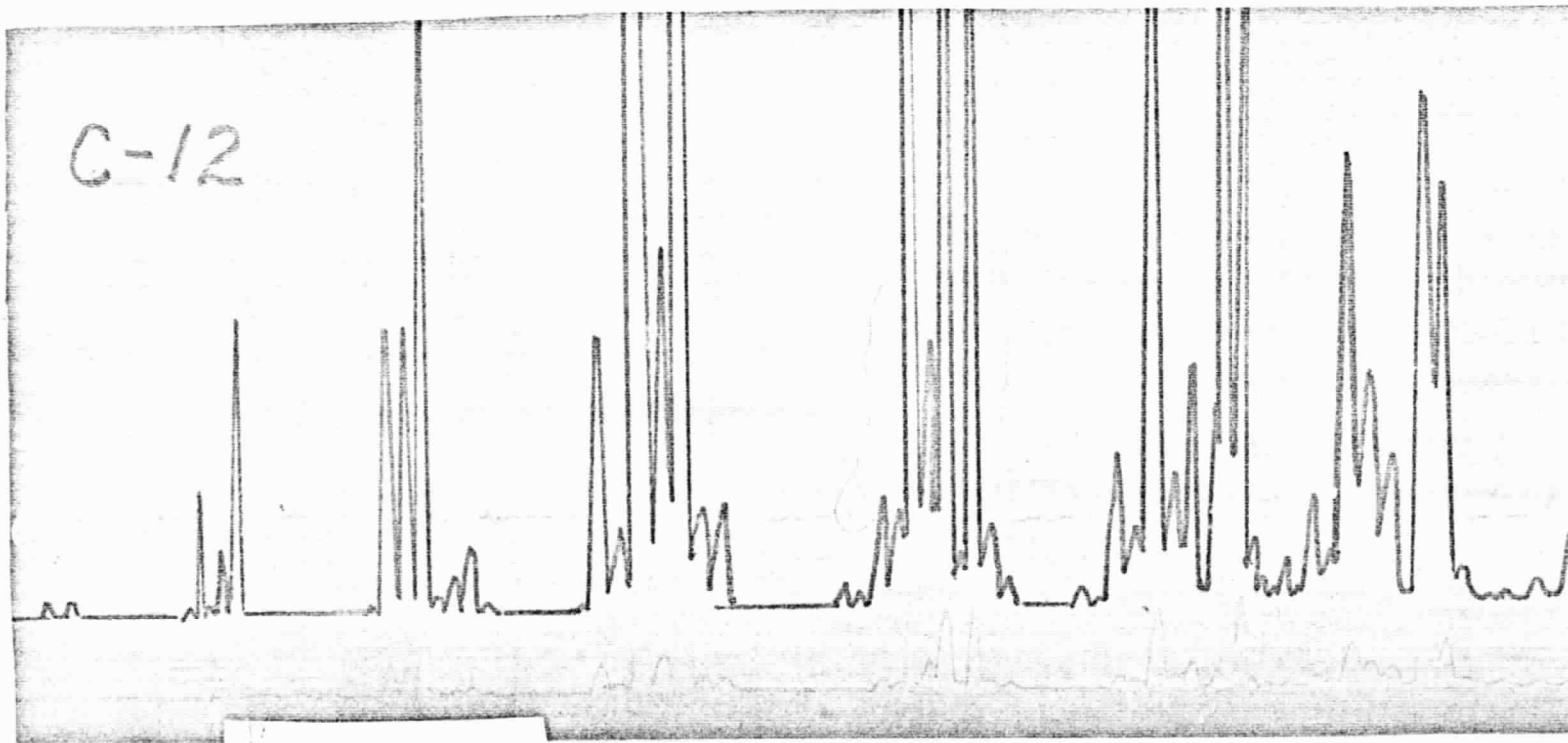
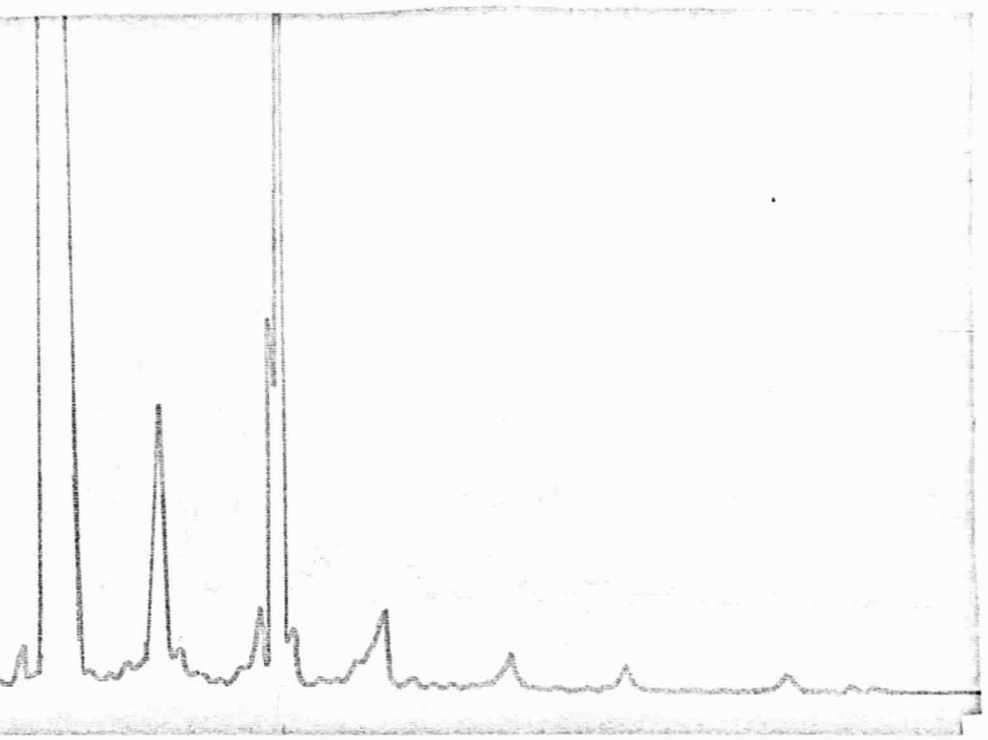
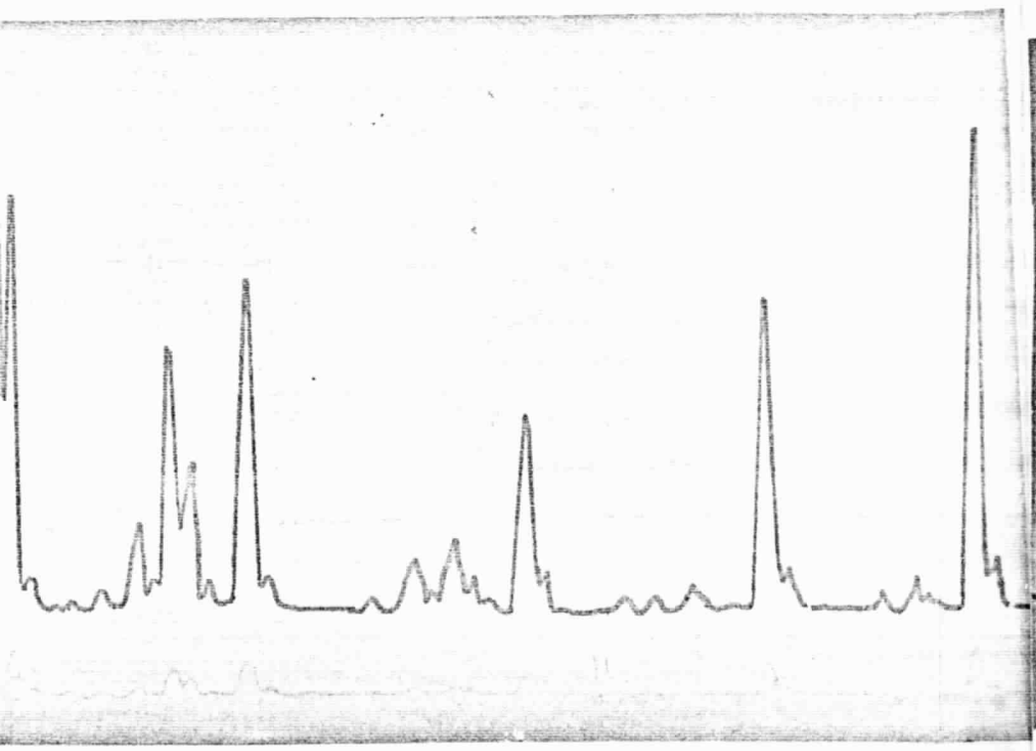


Figure 3

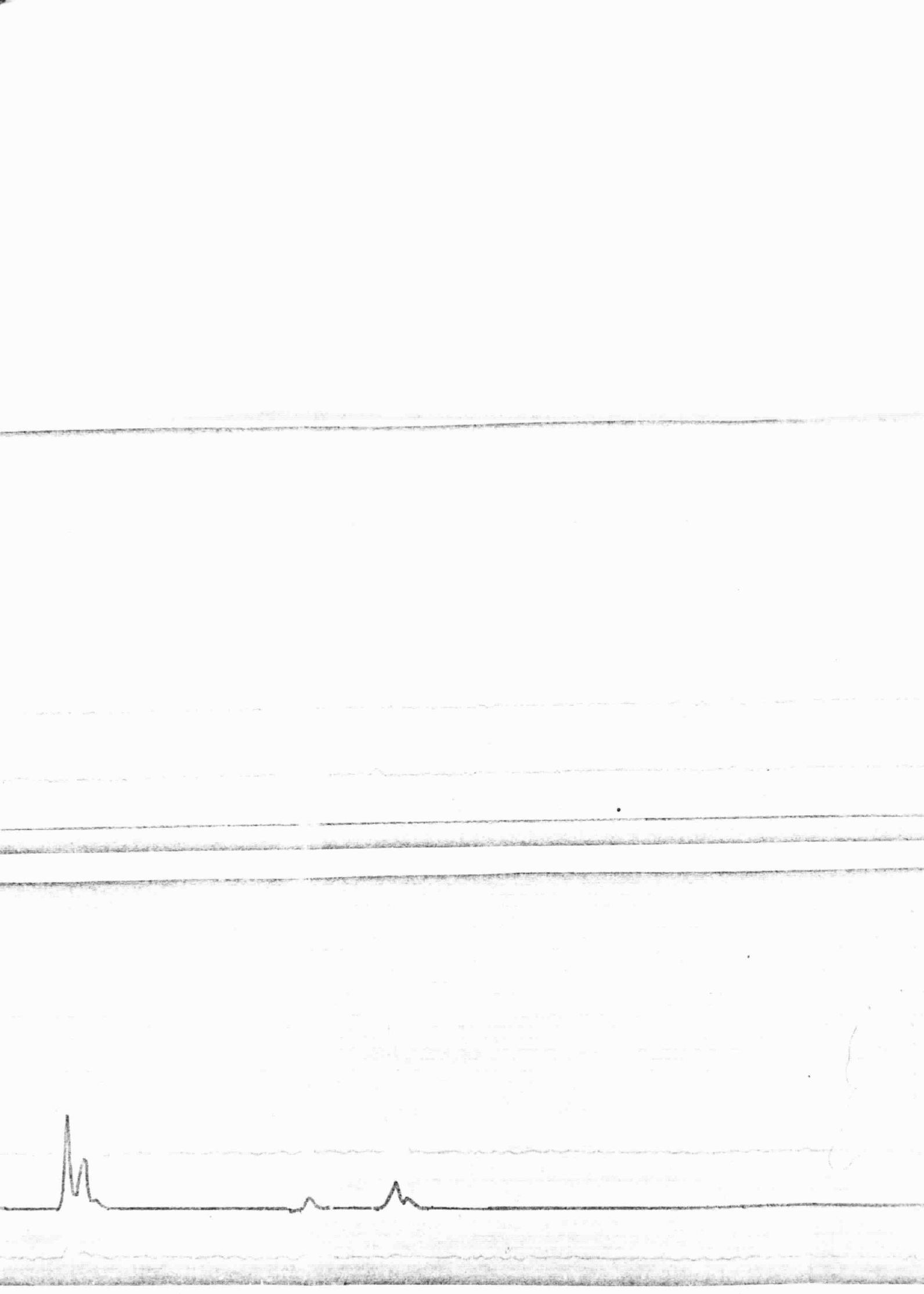
FOLDOUT FRAME. 1

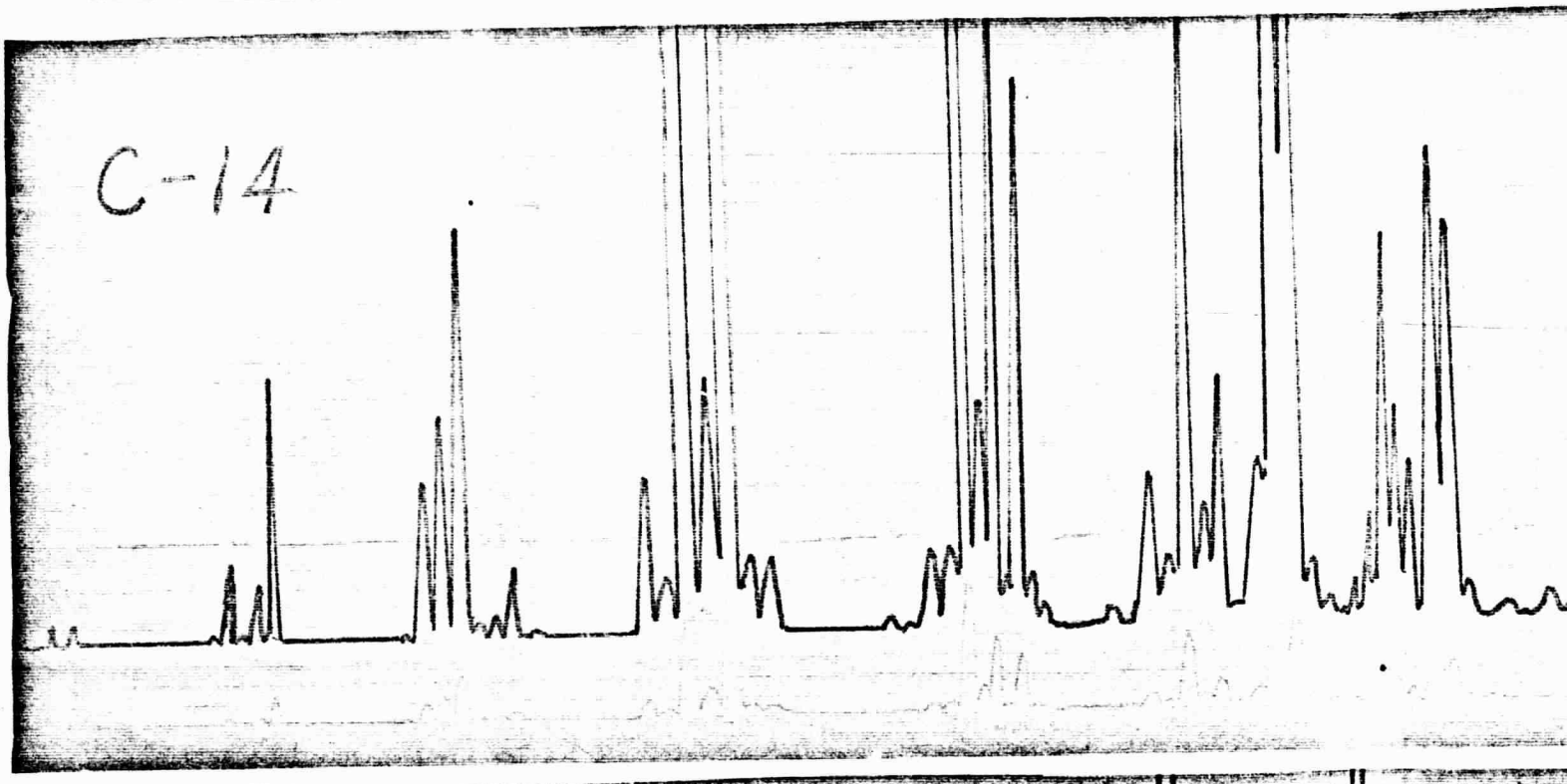


C-6



C-12





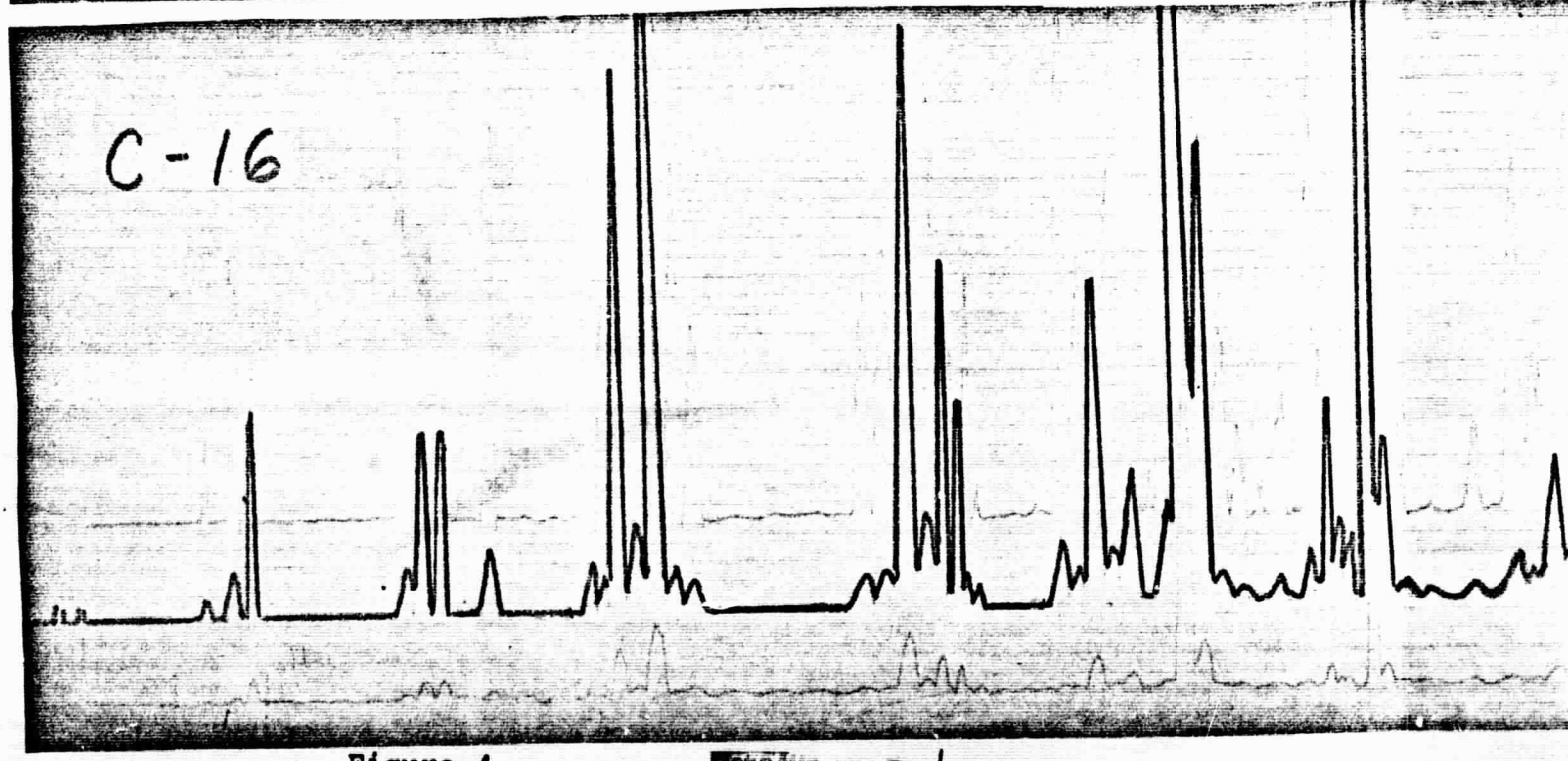
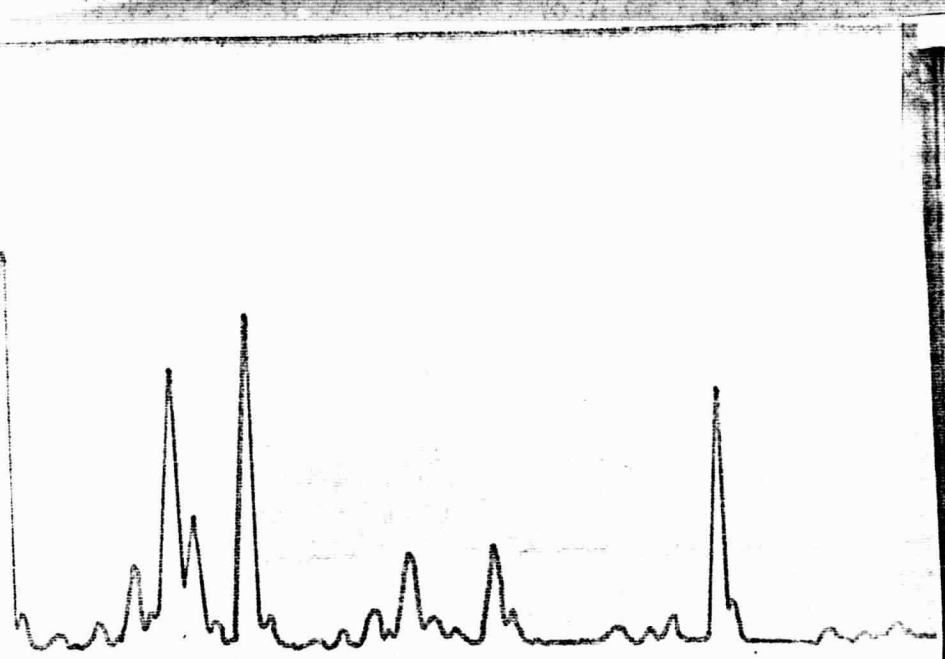
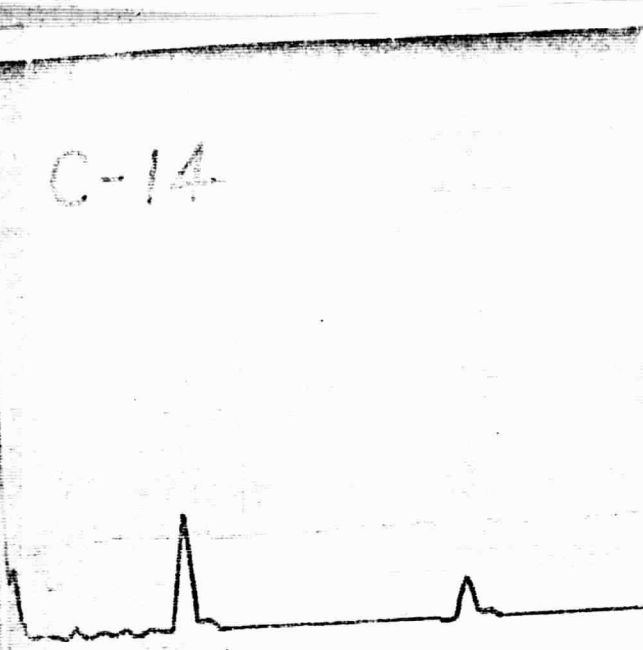
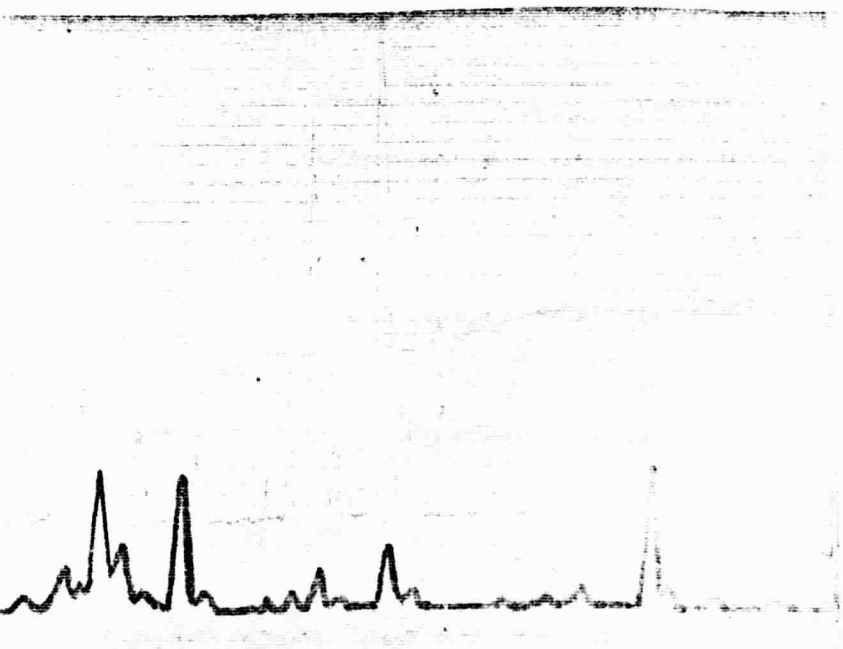


Figure 4

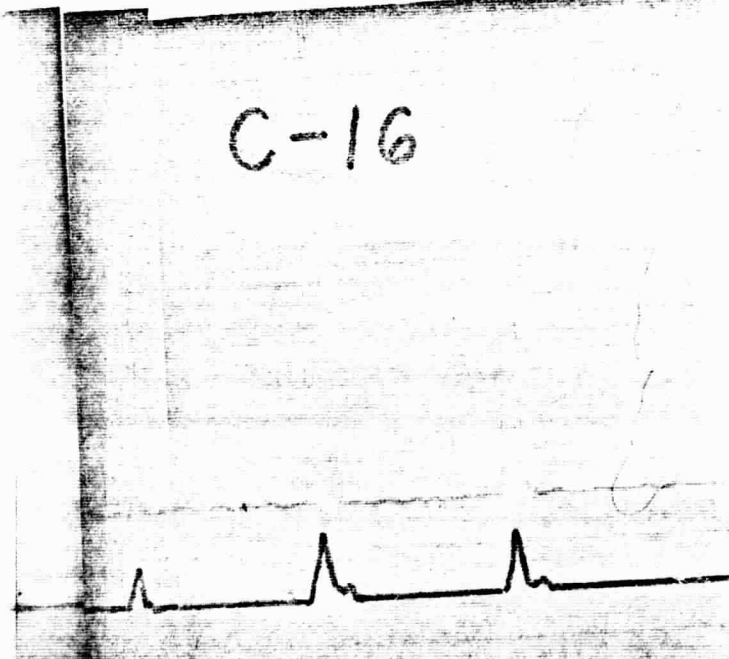


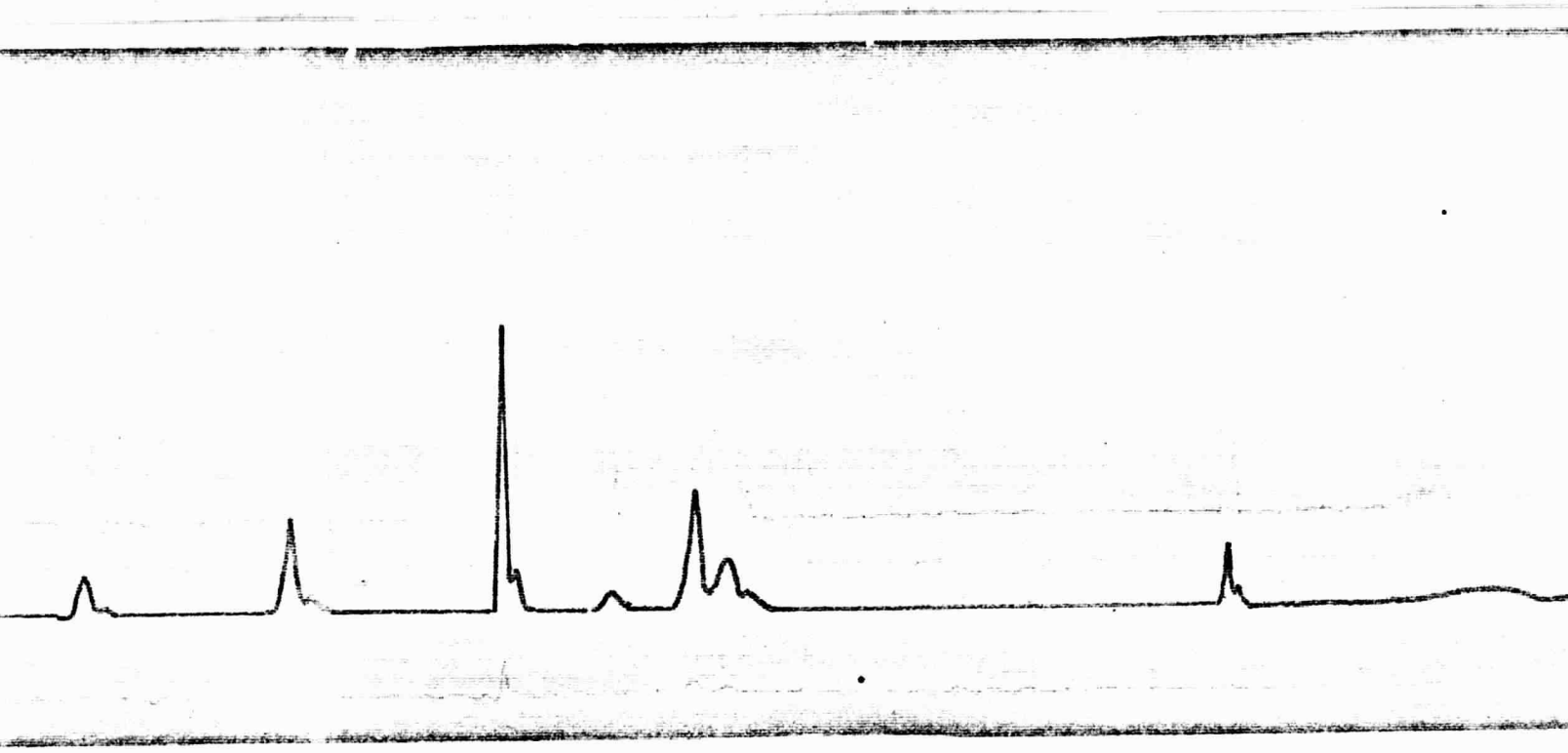
C-14

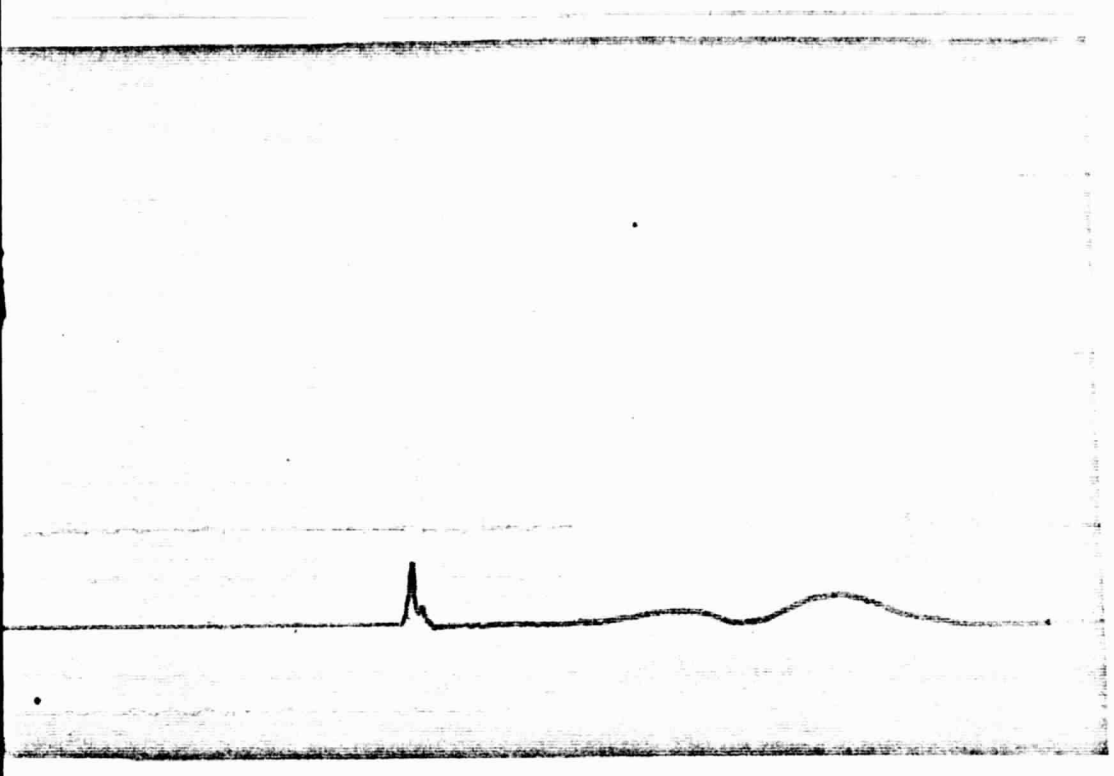


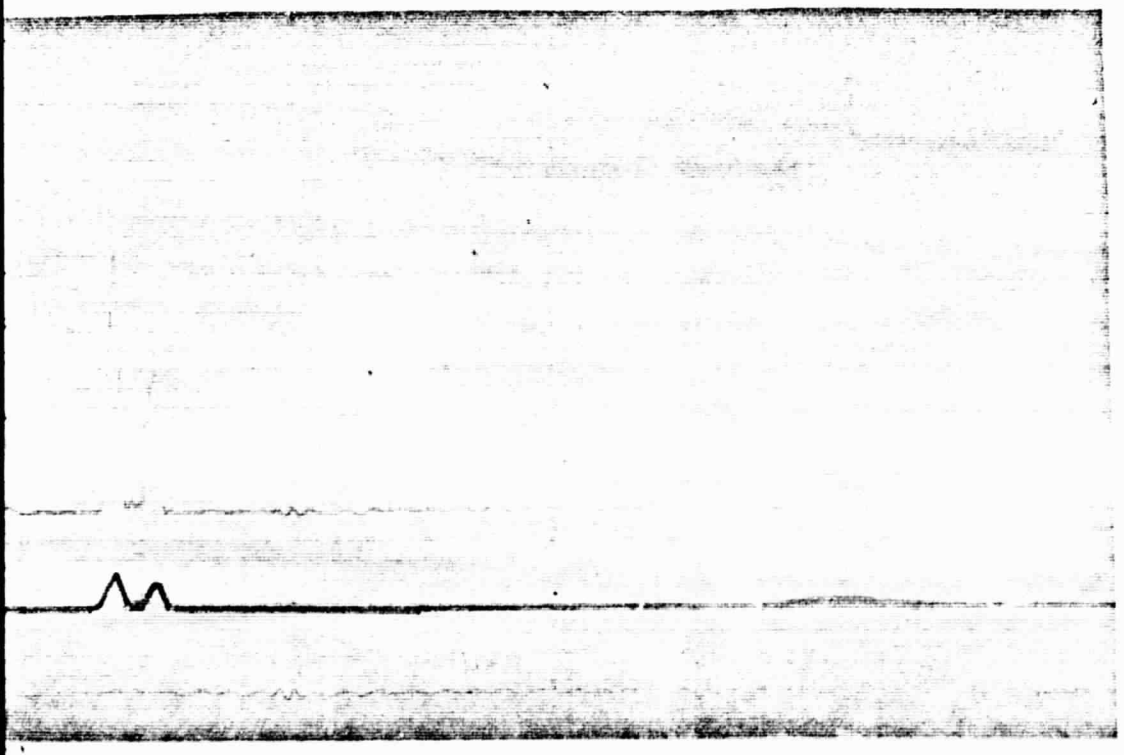


C-16









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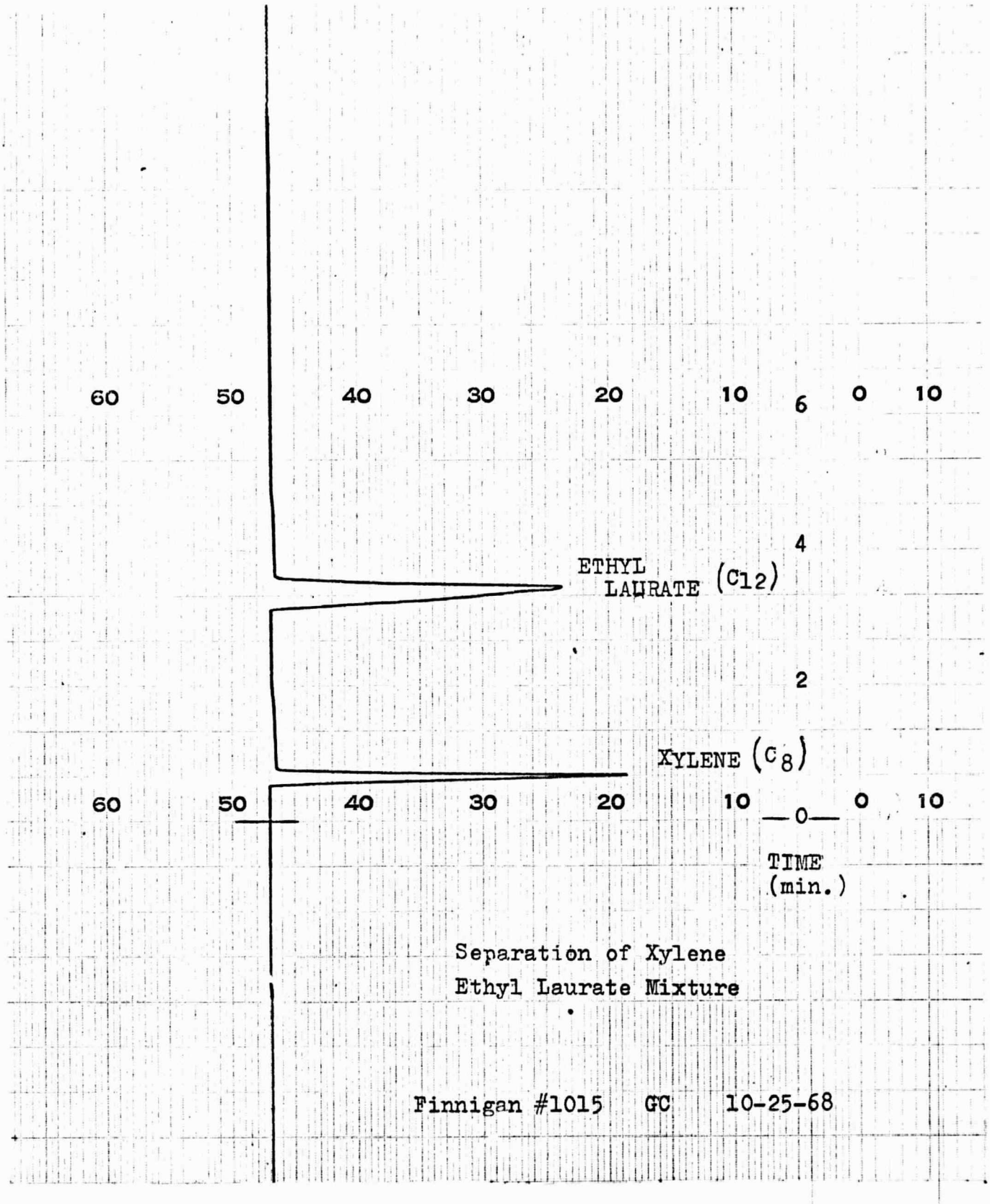
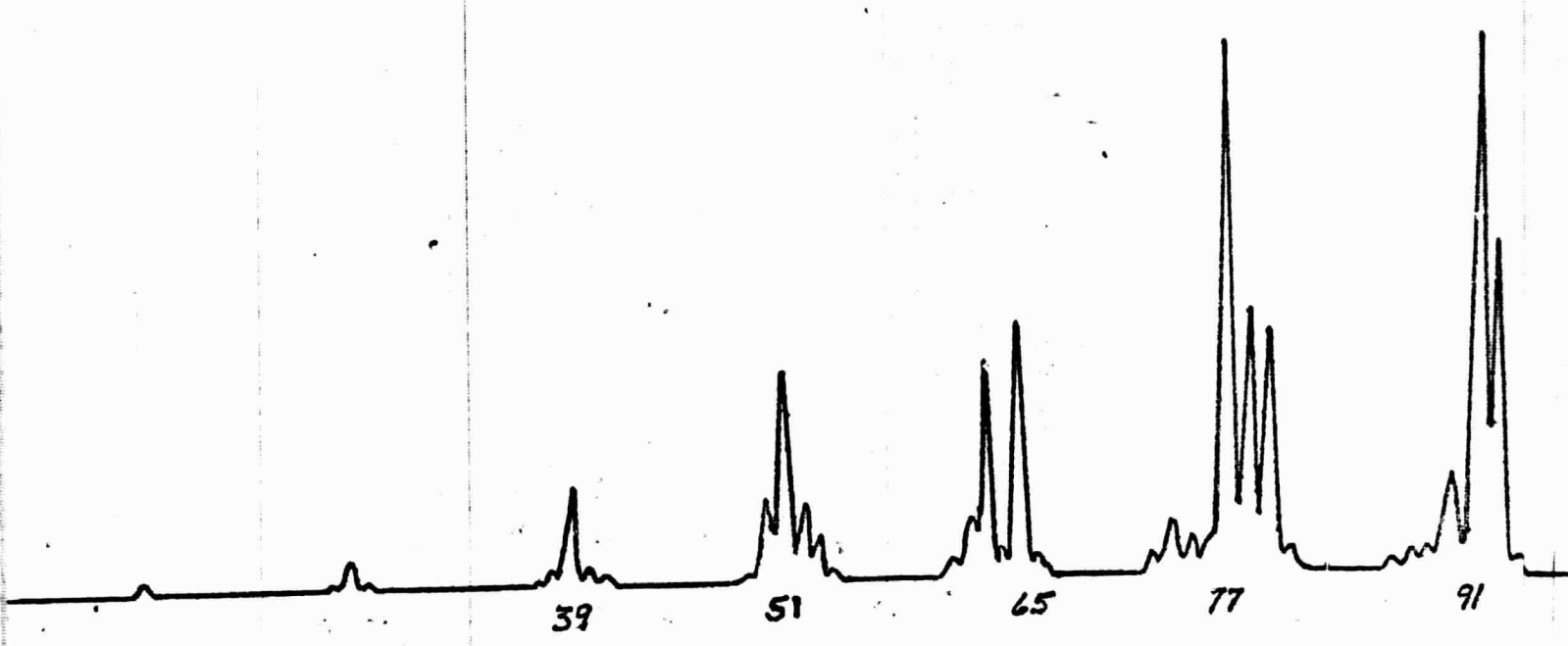
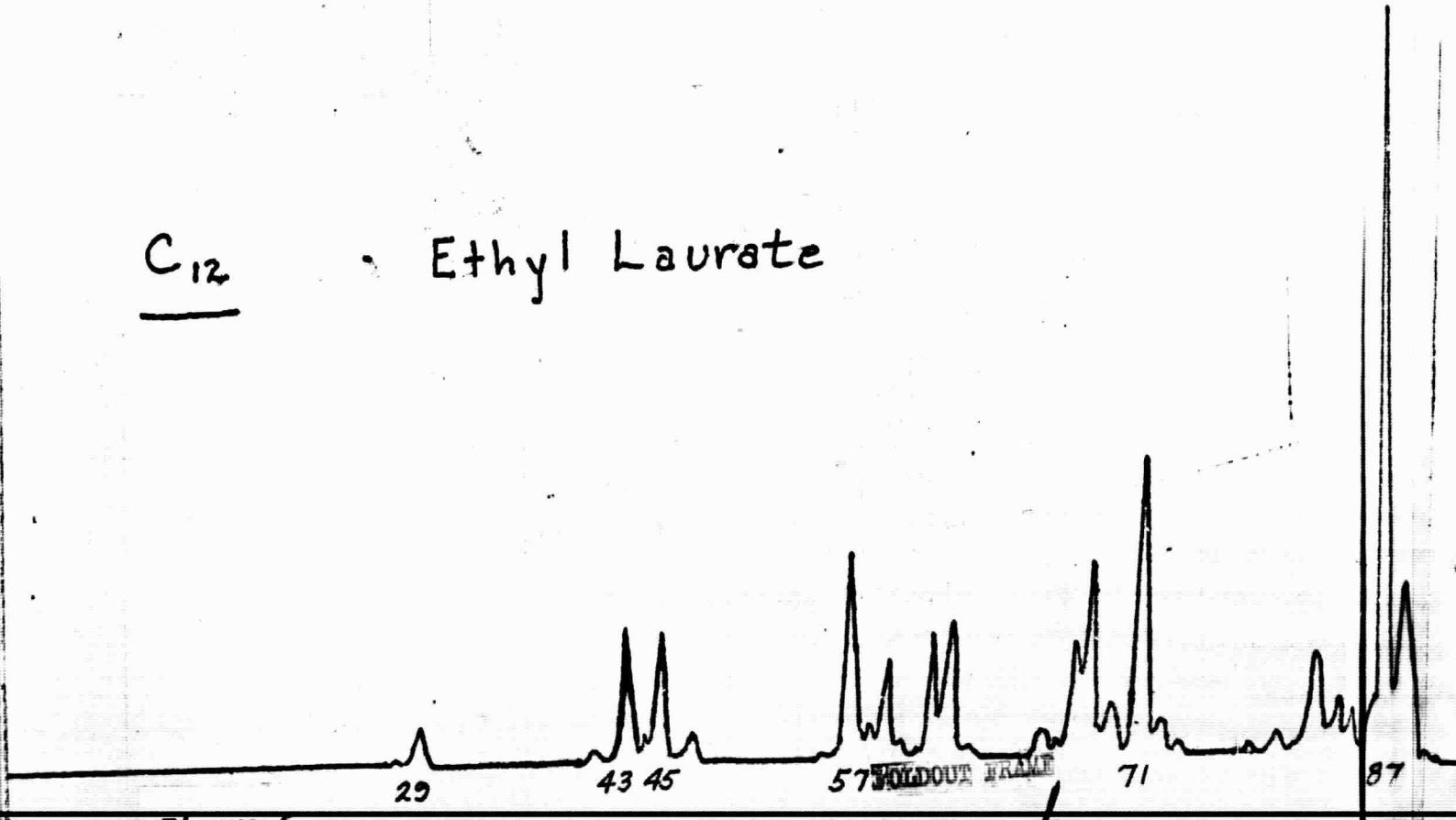

60
50
40
30
20
10
6
0
10
4
ETHYL
LAURATE (C12)
2
XYLENE (C8)
60
50
40
30
20
10
0
0
10
TIME
(min.)
Separation of Xylene
Ethyl Laurate Mixture
Finnigan #1015 GC 10-25-68

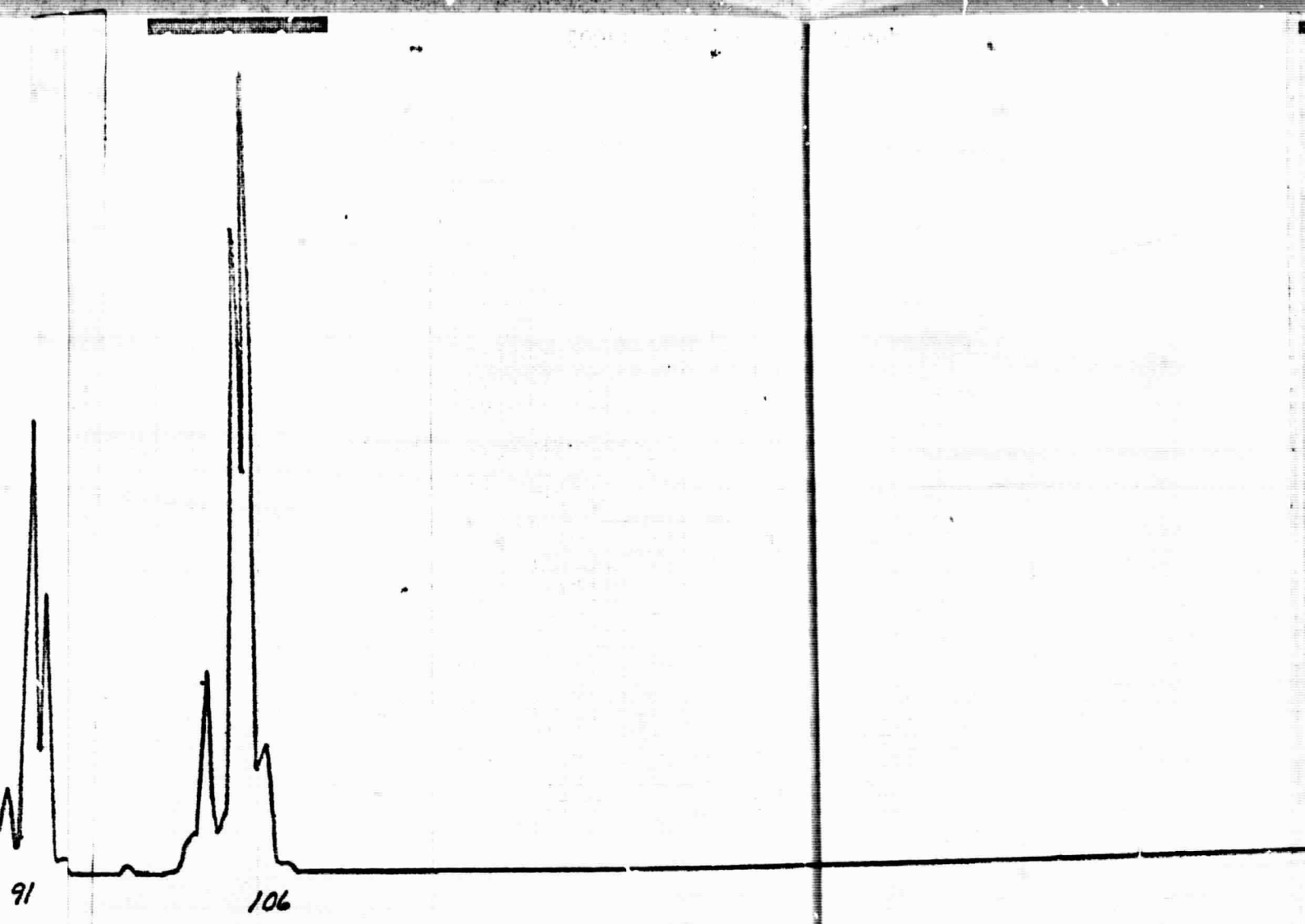
Figure 5

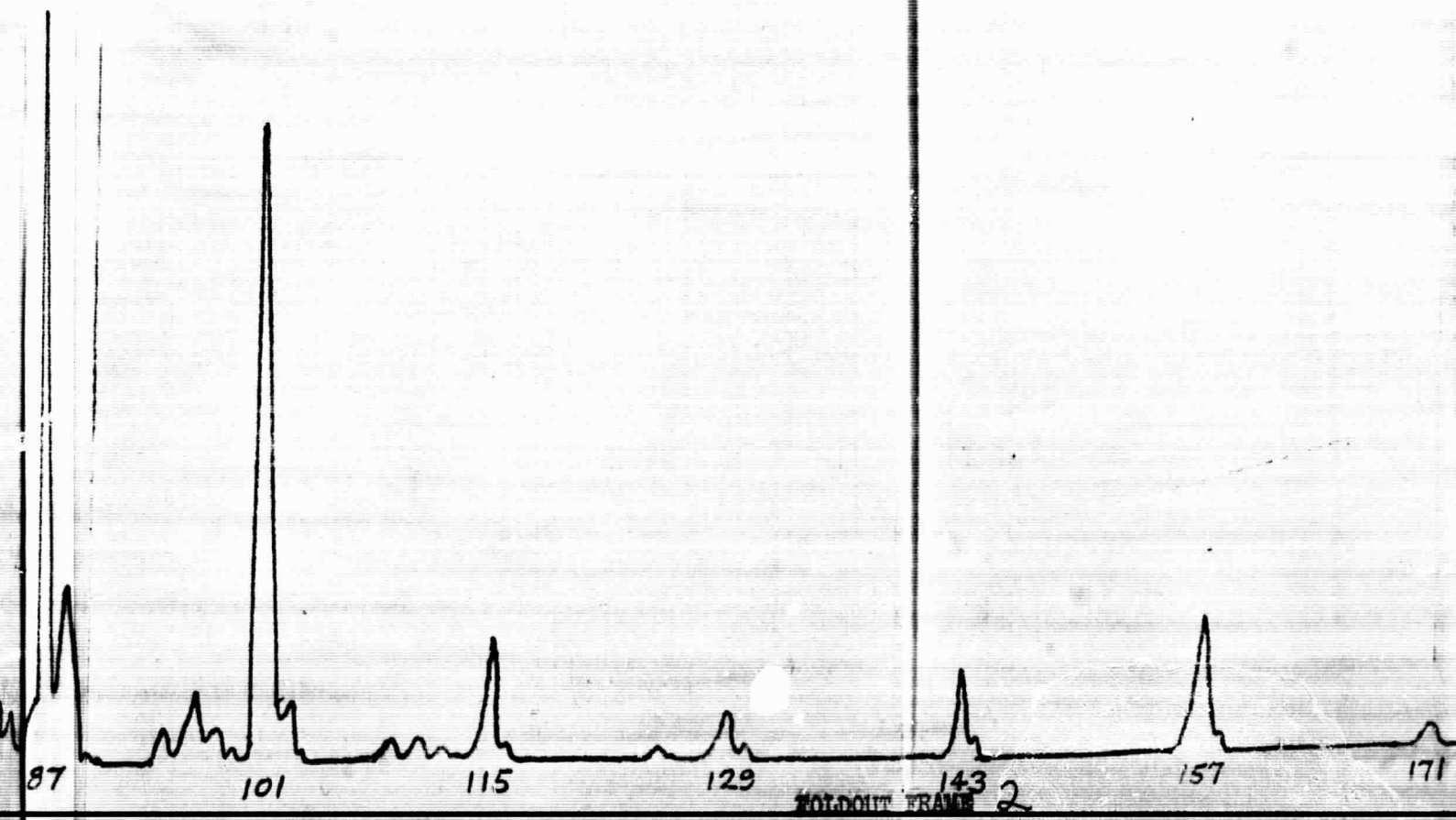
C_8 · m · Xylene



C_{12} · Ethyl Laurate







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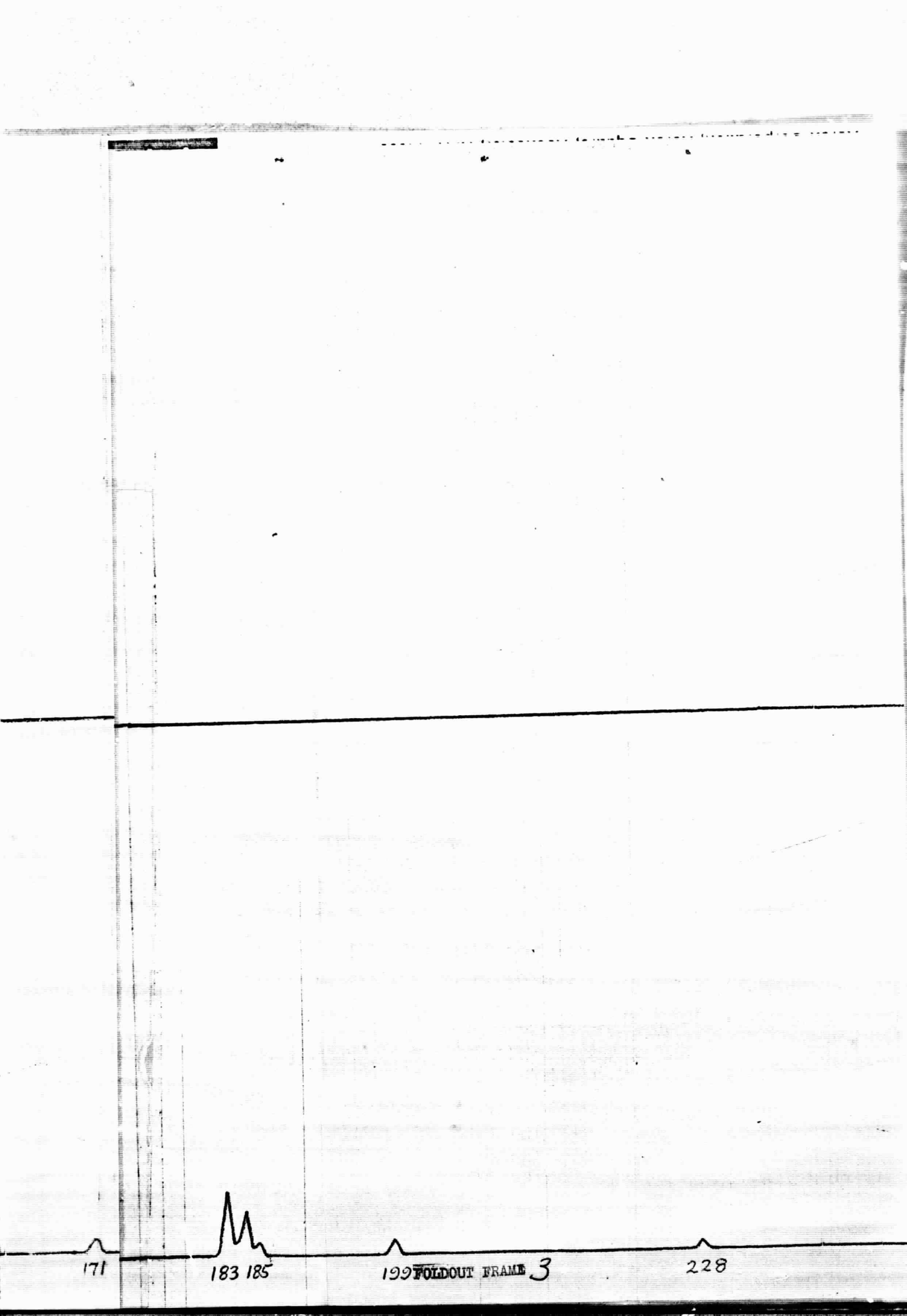
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183 185
199
FOLDOUT FRAME 3
228